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SECOND PERFORMANCE AUDIT OF INSPECTION AND MAINTENANCE CALIBRATION GASES

by

Robert S. Wright
Curtis E. Moore
Clifford E. Decker
Research Triangle Institute
Research Triangle Park, NC 27709

William F. Barnard
Environmental Monitoring Systems Laboratory
U.S. Environmental Protection Agency
Research Triangle Park, NC 27711

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EPA Project Officers
William F. Barnard
Darryl von Lehmden

ENVIRONMENTAL MONITORING SYSTEMS LABORATORY
OFFICE OF RESEARCH AND DEVELOPMENT
U.S. ENVIRONMENTAL PROTECTION AGENCY
RESEARCH TRIANGLE PARK, NC 27711

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16. ABSTRACT

The purpose of the audit was to determine the accuracy of calibration gases being used by state and local inspection and maintenance (I/M) programs. Forty-six calibration gas cylinders were borrowed from 16 representative I/M programs. These cylinders had been prepared by six different specialty gas producers. Typically, they contained propane (or hexane), carbon monoxide, and carbon dioxide in nitrogen. Research Triangle Institute (RTI) analyzed these calibration gases using nondispersive infrared photometry and gas chromatography with flame ionization and thermal conductivity detectors. NBS gaseous Standard Reference Materials were the reference standards for the analyses. Seventy-three percent of the certified hydrocarbon, carbon monoxide, and carbon dioxide concentrations were found to be accurate to within ± 2 percent relative. Ninety percent of the certified concentrations were found to be accurate to within ± 5 percent relative. The six I/M calibration gases that had been prepared and analyzed according to the EPA recommended practice were accurate to within ± 2 percent relative. These accuracies are similar to those found in a 1985 RTI audit of I/M calibration gases purchased directly from specialty gas producers and in a 1985 RTI audit of sulfur dioxide, nitric oxide, and carbon monoxide EPA Protocol Gases. Confirmatory analyses of 12 of the 46 cylinders at two EPA laboratories showed good agreement with RTI's analyses for carbon monoxide and carbon dioxide and fair agreement for the hydrocarbon analyses.

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SECOND PERFORMANCE AUDIT OF INSPECTION AND MAINTENANCE CALIBRATION GASES

Robert S. Wright, Curtis E. Moore, and Clifford E. Decker
Research Triangle Institute
Research Triangle Park, North Carolina

William F. Barnard
U.S. Environmental Protection Agency
Research Triangle Park, North Carolina

The purpose of the audit was to determine the accuracy of calibration gases being used by state and local inspection and maintenance (I/M) programs. Forty-six calibration gas cylinders were borrowed from 16 representative I/M programs. These cylinders had been prepared by six different specialty gas producers. Typically, they contained propane (or hexane), carbon monoxide, and carbon dioxide in nitrogen. Research Triangle Institute (RTI) analyzed these calibration gases using nondispersive infrared photometry and gas chromatography with flame ionization and thermal conductivity detectors. NBS gaseous Standard Reference Materials were the reference standards for the analyses. Seventy-three percent of the certified hydrocarbon, carbon monoxide, and carbon dioxide concentrations were found to be accurate to within $\pm$ 2 percent relative. Ninety percent of the certified concentrations were found to be accurate to within $\pm$ 5 percent relative. The six I/M calibration gases that had been prepared and analyzed according to the EPA recommended practice were accurate to within + 2 percent relative. These accuracies are similar to those found in a 1985 RTI audit of I/M calibration gases purchased directly from specialty gas producers and in a 1985 RTI audit of sulfur dioxide, nitric oxide, and carbon monoxide EPA Protocol Gases. Confirmatory analyses of 12 of the 46 cylinders at two EPA laboratories showed good agreement with RTI's analyses for carbon monoxide and carbon dioxide and fair agreement for the hydrocarbon analyses.

INTRODUCTION

The Clean Air Act Amendments of 1977 provide for the establishment of motor vehicle inspection and maintenance (I/M) programs for states or particular areas within a state that do not meet the National Ambient Air Quality Standard for carbon monoxide and/or ozone. The inspection function of an I/M program identifies those motor vehicles whose carbon monoxide and/or hydrocarbon tailpipe emissions exceed some specified limit. Accurately calibrated analytical instrumentation must be used to measure these emissions. The U.S. Environmental Protection Agency's (EPA's) Emission Control System Performance Warranty regulations specify that the instrumentation must be calibrated with gas traceable to National Bureau of Standards (NBS) standards to within $\pm$ 2 percent within a week of the emissions measurements (1).

EPA has established a recommended practice for naming I/M calibration gas (2,3). This practice contains procedures that specialty gas producers can use to blend, analyze, and label calibration gases for the inspection analyzers. Using the practice, the producers should be able to attain $\pm$ 2 percent accuracy. The practice states the following in a section titled "EPA Audits":

> "EPA does not think it is necessary to check samples of a batch of calibration gas named according to the Recommended Practice prior to the sale and delivery of cylinders of the batch. However, EPA may from time-to-time order gas mixtures named according to the Recommended Practice from manufacturers through a third party for audit purposes. The results of such audits are likely to be published for public inspection."

In 1985, Research Triangle Institute (RTI) conducted the first audit of I/M calibration gases under EPA contract (4). In that audit, I/M calibration gases were purchased directly from 13 specialty gas producers via a third party buyer. These gases were specified to contain 640 parts per million (ppm) propane and 1.6 percent carbon monoxide in nitrogen. They were specified to be prepared and analyzed according to the recommended practice in the three cases in which the producers could provide such a gas mixture. The highest accuracy available from each producer was specified in the 10 remaining cases. These gases were analyzed at RTI. Twelve of the 13 certified carbon monoxide concentrations and 9 of the 13 certified propane concentrations were found to be accurate to within $\pm$ 2 percent relative. Confirmatory analyses at two EPA laboratories showed good agreement with RTI's analyses.

This paper presents the results of a second performance audit of I/M calibration gases. A total of 46 gases were borrowed from 16 state and local I/M programs for this audit rather than purchasing them directly from the specialty gas producers. The characteristics of these gases are summarized in Table 1. The gases were prepared by six specialty gas producers. Generally, they contained propane (or hexane), carbon monoxide, and carbon dioxide in nitrogen. For four

cylinders, hexane was the hydrocarbon rather than propane. There was no carbon dioxide present in three of the other cylinders. The gases were analyzed and certified either by the specialty gas producer or by the I/M program or its contractor. It should be noted that only two I/M programs (Davis County, Utah and Salt Lake City-County, Utah) supplied gases that were prepared and analyzed according to the recommended practice.

ANALYTICAL PROCEDURES

After the 46 I/M calibration gases were shipped to RTI, their propane (or hexane), carbon monoxide, and carbon dioxide concentrations were measured at RTI. Subsequently, 12 of the gases were sent to EPA laboratories in Ann Arbor, Michigan and Research Triangle Park, North Carolina for confirmatory analyses. The gases were then returned to the I/M programs.

At RTI, propane (or hexane) and impurity hydrocarbon concentrations were measured using gas chromatography with flame ionization detection. Carbon monoxide concentrations were measured using gas chromatography with thermal conductivity detection. Carbon dioxide concentrations were measured using a nondispersive infrared analyzer. NBS gaseous Standard Reference Materials (SRMs) were used as the reference standards for the measurements. Groups of six I/M calibration gases and two reference standards were measured sequentially using a sampling manifold controlled by a digital timer. The instruments were calibrated "on the fly" from the reference standard measurements. Generally, three replicate measurements were made for each I/M calibration gas concentration.

At EPA-Ann Arbor, total hydrocarbon (as propane) concentrations were measured using a flame ionization detector. Carbon monoxide and carbon dioxide concentrations were measured using nondispersive infrared analyzers. The reference standards for these measurements were secondary standards that were traceable to NBS gaseous SRMs to within $\pm$ 1/2 percent relative. A single measurement was made for each I/M calibration gas concentration.

At EPA-Research Triangle Park, total hydrocarbon (as propane) concentrations were measured using a flame ionization detector. Carbon monoxide and carbon dioxide concentrations were measured using nondispersive infrared analyzers. The I/M calibration gases and reference standards were diluted with a digital static gas blender to bring the concentrations to within the instruments' operating ranges. The reference standards for these measurements were NBS gaseous SRMs. A single measurement was made for each I/M calibration gas concentration.

AUDIT RESULTS

A summary of RTI's measurements of the I/M calibration gas concentrations is given in Table 2 for hydrocarbons, in Table 3 for carbon monoxide, and in Table 4 for carbon dioxide. In each table, the specialty gas producers and the I/M programs are listed. Also listed

is the percent difference between the certified concentration and RTI's mean measured concentration for each I/M calibration gas. The percent difference is calculated using the following formula:

$$100 \left[\frac{\text{Certified Concentration - Mean Measured Concentration}}{\text{Mean Measured Concentration}} \right]$$

It should be noted that the certified propane (or hexane) concentrations were compared with RTI's measured propane (or hexane) concentrations when Table 2 was developed. RTI's measured impurity hydrocarbon concentrations were not included in the calculation.

Because three measurements were generally used to calculate the mean measured concentration, data are available to estimate the precision of RTI's measurements. The coefficient of variation (CV) is used as an index of precision where

$$CV = 100 \left[\text{Standard Deviation/Mean Concentration} \right]$$

The CVs for RTI's measured hydrocarbon concentrations ranged from 0.0 to 2.1 percent relative with a mean CV of 0.4 percent relative. The corresponding carbon monoxide CVs had a range from 0.0 to 4.4 percent relative and had a mean value of 0.5 percent relative. The CVs for the carbon dioxide data were more precise with a range from 0.0 to 0.3 percent relative and a mean value of 0.1 percent relative. Generally, a particular I/M calibration gas was reanalyzed if its CV was larger than 0.5 percent relative.

RTI's measurements are further summarized in the following table which gives the number of I/M calibration gases falling within a given range of absolute values of percent differences:

Component	0 to 2 Percent Relative	2 to 5 Percent Relative	5 to 10 Percent Relative	>10 Percent Relative
Hydrocarbon	31	9	2	4
Carbon Monoxide	36	6	1	3
Carbon Dioxide	32	7	4	0
TOTAL	99	22	7	7

For the combined hydrocarbon, carbon monoxide, and carbon dioxide data, 73 percent of the absolute values of the percent differences fell between 0 and 2 percent relative. Ninety percent of the absolute values fell between 0 and 5 percent relative. These percentages are similar to those which were obtained in the first audit of I/M calibration gases (i.e., 81 and 88 percent of the gases, respectively). Additionally, these percentages are similar to those which were obtained in a 1985 RTI audit of sulfur dioxide, nitric oxide, and carbon monoxide EPA Protocol Gases (i.e., 70 and 94 percent of the gases, respectively)(5). It should be noted that all I/M calibration gases that had been prepared and analyzed according to the EPA recommended practice were accurate to within $\pm$ 2 percent relative.

EPA CONFIRMATORY ANALYSES

Following RTI's measurements, 12 of the 46 I/M calibration gases were sent to EPA laboratories in Ann Arbor (EPA-AA) and Research Triangle Park (EPA-RTP) for confirmatory analyses. The purpose of these confirmatory analyses was to determine whether RTI's measurements were accurate for these 12 cylinders and, by inference, for the remaining 34 cylinders. Table 5 is a summary of the comparison between RTI's measurements and EPA's confirmatory analyses. In this table, the I/M programs are listed as are the percent differences between each EPA laboratory's and RTI's measured concentrations. It should be noted that the EPA total hydrocarbon concentrations are being compared with the sum of RTI propane (or hexane) and impurity hydrocarbon concentrations in those cases in which RTI detected impurities. The confirmatory analyses are further summarized in the following table which gives the number of I/M calibration gases falling within a given range of absolute values of percent differences:

EPA Lab	Component	0 to 2 Percent Relative	2 to 5 Percent Relative	5 to 10 Percent Relative	> 10 Percent Relative
EPA-AA	Hydrocarbon	10	2	0	0
	Carbon Monoxide	11	1	0	0
	Carbon Dioxide	10	0	0	0
EPA-RTP	Hydrocarbon	8	3	1	0
	Carbon Monoxide	5	3	4	0
	Carbon Dioxide	10	0	0	0

In general, good agreement among the three sets of measurements was seen for carbon monoxide and carbon dioxide. However, the carbon monoxide measurements by EPA-RTP exhibited some problems associated with the dilution of samples prior to analysis. Only fair agreement was seen for the hydrocarbon measurements. The agreement between EPA-RTP's and RTI's hexane measurements was not good. Better agreement had been seen for propane and carbon monoxide during the first audit of I/M calibration gases.

CONCLUSIONS

Any performance audit is an instantaneous snapshot of the process being audited. These audit results should not be regarded as a final statement concerning the accuracy of I/M calibration gases. They can be used as an indicator of the current status of the field as a whole. Focusing on individual results may not be as valid as inspecting their collective characteristics.

RTI's analyses have found that 73 percent of the I/M calibration gases' hydrocarbon, carbon monoxide, and carbon dioxide concentrations were accurate to within $\pm$ 2 percent relative using NBS Standard Reference Materials (SRMs) as the reference standards. This percentage is

similar to that found for the first audit of I/M calibration gases and for a recent audit of EPA Protocol Gases. The confirmatory analyses by the two EPA laboratories support RTI's measured concentrations for 12 of the 46 calibration gases. Good agreement among the three sets of measurements was seen for carbon monoxide and carbon dioxide. Only fair agreement was seen for the hydrocarbon measurements.

Several aspects of the analytical procedures used in the audit should be investigated for possible improvement. The confirmatory hydrocarbon analyses suggest that there is an unknown source or error or imprecision in RTI's and/or EPA's instrumentation. The carbon monoxide measurements at EPA-RTP suggest that dilution of the I/M calibration gases prior to their analysis may add uncertainty to the final results.

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TABLE 1. I/M CALIBRATION GASES ANALYZED IN THE SECOND PERFORMANCE AUDIT

Specialty Gas Producer	I/M Program	No. of Cyls.	Cyl. Mat.(1)	Certified Concentration		
				Hydrocarbon (ppm)	Carbon Monoxide(%)	Carbon Dioxide(%)
Airco (2)	Nevada	2	Al	801, 2544	1.60, 8.00	0.0
Airco	Oregon (2)	3	Al	800	2.	13.
Air. Prod. (2)	Memphis, TN	3	DS	3000	8.	14.5
Liq. Carb.	Arizona (2)	2	Al	300(3),800(3)	1.50, 6.0	5.2
Liq. Carb. (2)	Michigan	3	Al	600	1.6	11.
Liq. Carb.	Wisconsin (2)	3	Al	443-473	1.17-1.20	4.98-5.00
MG Sci. (2)	Nashville, TN	3	DS	209(3), 446	2.34-2.49	9.47-9.50
Sci. Gas. Prod.(2)	Davis Co., UT	3	DS	606(4)	1.62(4)	11.1(4)
Scott Spec. (2)	California	3	Al	600	1.60	11.0
Scott Spec.	Colorado(2)	3	DS	601-3131	1.54-8.24	0.0-14.14
Scott Spec.	Connecticut(2)	3	Al	261-457	1.00-1.45	10.87-10.93
Scott Spec. (2)	New Jersey	3	Al	604-608	1.60-1.62	10.94-11.16
Scott Spec. (2)	New York	3	Al	2993	7.98	16.16
Scott Spec. (2)	Pennsylvania	3	Al	602-608	1.59-1.62	10.90-11.02
Scott Spec. (2)	Salt Lake, UT	3	DS	600-602(4)	1.60(4)	10.98(4,5)
Scott Spec.	Washington(2)	3	Al	1599-1601	4.02-4.03	4.6-4.7

(1) Cylinder Material: Al = Aluminum, DS = Disposable Steel.
(2) Certifying Analyst.
(3) The hydrocarbon is hexane rather than propane.
(4) Prepared and analyzed according to the EPA recommended practice.
(5) Producer's unanalyzed concentration.

TABLE 2. SUMMARY OF RESULTS FOR RTI HYDROCARBON MEASUREMENTS IN THE SECOND PERFORMANCE AUDIT

Specialty Gas Producer	I/M Program	Percent Difference(1) 1st Cylinder	2nd Cylinder	3rd Cylinder
Airco (2)	Nevada	1.7	0.1	----
Airco	Oregon (2)	15.9	12.4	14.6
Air. Prod. (2)	Memphis, TN	4.2	4.3	-1.6
Liq. Carb.	Arizona (2)	3.3(3)	3.9(3)	----
Liq. Carb. (2)	Michigan	2.3	1.6	2.4
Liq. Carb.	Wisconsin (2)	2.5	1.3	3.0
MG Sci. (2)	Nashville, TN	-9.1(3)	6.3	-11.4(3)
Sci. Gas. Prod.(2)	Davis Co., UT	0.0(4)	0.0(4)	-0.1(4)
Scott Spec. (2)	California	-0.4	-0.2	-0.1
Scott Spec.	Colorado(2)	-0.2	-0.8	0.0
Scott Spec.	Connecticut(2)	-0.7	-0.5	-0.5
Scott Spec. (2)	New Jersey	0.2	-0.2	1.0
Scott Spec. (2)	New York	-1.0	-1.1	-1.0
Scott Spec. (2)	Pennsylvania	-0.8	-0.8	0.8
Scott Spec. (2)	Salt Lake, UT	-0.7(4)	-0.6(4)	-0.4(4)
Scott Spec.	Washington(2)	0.0	-0.4	0.4

(1) Percent difference between certified and mean RTI-measured concentrations = 100[(Certified Concentration-Measured Concentration)/Measured Concentration].
(2) Certifying Analyst.
(3) The hydrocarbon is hexane rather than propane.
(4) Prepared and analyzed according to the EPA recommended practice.

TABLE 3. SUMMARY OF RESULTS FOR RTI CARBON MONOXIDE MEASUREMENTS IN THE SECOND PERFORMANCE AUDIT

Specialty Gas Producer	I/M Program	Percent Difference(1) 1st Cylinder	2nd Cylinder	3rd Cylinder
Airco (2)	Nevada	-1.4	-0.1	----
Airco	Oregon (2)	11.5	11.5	10.6
Air. Prod. (2)	Memphis, TN	2.9	3.1	-0.8
Liq. Carb.	Arizona (2)	1.5	0.5	----
Liq. Carb. (2)	Michigan	0.2	0.4	0.4
Liq. Carb.	Wisconsin (2)	-1.3	-1.1	-1.6
MG Sci. (2)	Nashville, TN	-0.6	-7.2	-1.3
Sci. Gas. Prod.(2)	Davis Co., UT	-0.5(3)	-1.2(3)	-0.8(3)
Scott Spec. (2)	California	-2.6	-2.6	-2.7
Scott Spec.	Colorado(2)	1.0	2.1	0.6
Scott Spec.	Connecticut(2)	0.8	-0.5	0.9
Scott Spec. (2)	New Jersey	-0.1	0.0	0.2
Scott Spec. (2)	New York	-0.3	-0.3	0.2
Scott Spec. (2)	Pennsylvania	0.8	-0.4.	-0.4
Scott Spec. (2)	Salt Lake, UT	0.6(3)	0.9(3)	-0.4(3)
Scott Spec.	Washington(2)	-0.7	-0.6	-1.3

(1) Percent difference between certified and mean RTI-measured concentrations = $100\left[\text{(Certified Concentration-Measured Concentration)/Measured Concentration}\right]$.
(2) Certifying Analyst.
(3) Prepared and analyzed according to the EPA recommended practice.

TABLE 4. SUMMARY OF RESULTS FOR RTI CARBON DIOXIDE MEASUREMENTS IN THE SECOND PERFORMANCE AUDIT

Specialty Gas Producer	I/M Program	Percent Difference (1)		
		1st Cylinder	2nd Cylinder	3rd Cylinder
Airco (2)	Nevada	(3)	(3)	----
Airco	Oregon (2)	7.0	7.3	6.9
Air. Prod. (2)	Memphis, TN	3.0	3.1	-0.7
Liq. Carb.	Arizona (2)	0.0	0.0	----
Liq. Carb. (2)	Michigan	-1.0	-0.7	-0.7
Liq. Carb.	Wisconsin (2)	-0.7	-1.0	-1.0
MG Sci. (2)	Nashville, TN	-1.0	-0.3	-1.2
Sci. Gas. Prod.(2)	Davis Co., UT	-0.3(4)	-0.2(4)	-0.3(4)
Scott Spec. (2)	California	-2.6	-2.7	-2.7
Scott Spec.	Colorado(2)	(3)	3.2	2.3
Scott Spec.	Connecticut(2)	-1.8	-5.1	-1.6
Scott Spec. (2)	New Jersey	-0.9	-0.1	-0.1
Scott Spec. (2)	New York	-1.5	-1.2	-1.5
Scott Spec. (2)	Pennsylvania	-0.6	-0.9	-0.1
Scott Spec. (2)	Salt Lake, UT	-0.1(4,5)	-0.1(4,5)	-0.2(4,5)
Scott Spec.	Washington(2)	-1.4	1.2	0.9

(1) Percent difference between certified and mean RTI-measured concentrations = 100[(Certified Concentration-Measured Concentration)/Measured Concentration].
(2) Certifying Analyst.
(3) No carbon dioxide is present in this cylinder.
(4) Prepared and analyzed according to the EPA recommended practice.
(5) These values are relative to the producer's unanalyzed concentration.

TABLE 5. SUMMARY OF COMPARISON BETWEEN RTI MEASUREMENTS AND EPA CONFIRMATORY ANALYSES

	Percent Difference (1)					
	Hydrocarbons		Carbon Monoxide		Carbon Dioxide	
I/M Program	EPA-AA	EPA-RTP	EPA-AA	EPA-RTP	EPA-AA	EPA-RTP
Arizona	1.5(2)	-9.0(2)	-0.2	0.7	0.0	0.4
Arizona	2.2(2)	-3.4(2)	1.7	6.4(3)	0.1	0.1
California	1.7	-0.5	-0.3	0.3	-0.5	-0.7
Connecticut	0.9	-2.6	-0.4	0.5	-0.1	-0.4
Michigan	3.0	0.5	-0.5	0.1	-0.6	-0.4
Nevada	-0.1	-0.3	0.4	2.1	(4)	(4)
Nevada	1.0	1.4	2.5	5.9(3)	(4)	(4)
New Jersey	2.0	0.0	-0.3	2.1	-0.2	-0.3
New York	2.0	-2.4	1.9	7.1(3)	-0.9	-1.6
Oregon	1.6	-1.2	-0.5	3.8	-0.6	-0.7
Washington	0.8	-1.0	0.7	5.6(3)	-0.1	0.3
Wisconsin	1.8	1.0	0.3	2.0	0.5	1.1
Mean	1.5	-1.5	0.4	3.0	-0.2	-0.2
	1.5(5)	-0.5(5)		1.4(6)		

(1) Percent difference = $100 \left[\frac{\text{EPA Measured Concentration-RTI Measured Concentration}}{\text{RTI Measured Concentration}}\right]$.

(2) Hydrocarbon is hexane rather than propane.

(3) These cylinders were diluted by a factor of 100 rather than by a factor of 20 for the remaining cylinders.

(4) These cylinders did not contain carbon dioxide.

(5) Including only those cylinders that contain propane.

(6) Including only those cylinders that were diluted by a factor of 20.